

=> d hist

(FILE 'USPAT' ENTERED AT 07:17:20 ON 20 FEB 96)

L1 18 S LEUCO (2A) INDIGO  
L2 0 S HYDROGENATE# (2A) INDIGO  
L3 0 S HYDROGENATE# (2A) VAT DYE#  
L4 0 S HYDROGENATION (2A) (INDIGO OR VAT DYE#)

FILE 'JPOABS' ENTERED AT 07:23:32 ON 20 FEB 96

L5 3 S L1  
L6 0 S L4  
L7 0 S L3  
L8 0 S L2

FILE 'CA' ENTERED AT 07:26:40 ON 20 FEB 96  
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FILE COVERS 1967 - 14 Feb 1996 (960214/ED) VOL 124 ISS 8

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Roles are now available from 1967 to date.

=> s indigo (2a) hydrogenat?

1676 INDIGO  
132870 HYDROGENAT?

L1 2 INDIGO (2A) HYDROGENAT?

=> d all 1-2

L1 ANSWER 1 OF 2 CA COPYRIGHT 1996 ACS  
AN 122:163400 CA  
TI Method of dyeing cellulose-containing textiles with  
\*\*\*hydrogenated\*\*\* \*\*\*indigo\*\*\*  
IN Schnitzer, Georg; Suetsch, Franz; Schmitt, Michael; Kromm, Erich;  
Schlueter, Harald; Krueger, Rudolf; Weiper-Idelmann, Andreas  
PA BASF A.-G., Germany  
SO PCT Int. Appl., 18 pp.  
CODEN: PIXXD2  
PI WO 9423114 A1 941013  
DS W: AU, BR, BY, CA, CN, CZ, FI, HU, JP, KR, KZ, NO, NZ, PL, RU, UA,  
US  
RW: AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE  
AI WO 94-EP873 940321  
PRAI DE 93-4310122 930330  
DE 93-4315873 930512  
DT Patent  
LA German  
IC ICM D06P001-22  
ICS D06P005-20  
CC 40-6 (Textiles and Fibers)  
Section cross-reference(s): 61  
AB Title method comprises prepg. an aq. soln. of leucoindigo by  
catalytic \*\*\*hydrogenation\*\*\* of \*\*\*indigo\*\*\* and conversion  
of the leucoindigo, after absorption on the textile material, by

oxidn. by air to the pigment form. The redn. of indigo to leucoindigo by catalytic hydrogenation eliminates the use of inorg. S-contg. compds. and hydroxyacetone as reducing agents and eliminates the use of sugar stabilizers, thereby reducing the COD, TOC, and BOD in the wastewater.

ST indigo dyeing cellulosic catalytic hydrogenation; leucoindigo catalytic \*\*\*hydrogenation\*\*\* \*\*\*indigo\*\*\* dyeing; wastewater sulfur elimination \*\*\*indigo\*\*\* catalytic \*\*\*hydrogenation\*\*\*

IT Hydrogenation  
(catalytic; method of dyeing cellulose-contg. textiles with \*\*\*hydrogenated\*\*\* \*\*\*indigo\*\*\* )

IT Dyeing  
(of cellulose-contg. textiles with \*\*\*hydrogenated\*\*\* \*\*\*indigo\*\*\* )

IT 482-89-3, Indigo  
RL: RCT (Reactant)  
(method of dyeing cellulose-contg. textiles with hydrogenated)

IT 1310-73-2, Sodium hydroxide, uses 6537-68-4, Leucoindigo  
RL: NUU (Nonbiological use, unclassified); USES (Uses)  
(method of dyeing cellulose-contg. textiles with \*\*\*hydrogenated\*\*\* \*\*\*indigo\*\*\* )

L1 ANSWER 2 OF 2 CA COPYRIGHT 1996 ACS

AN 122:163396 CA

TI Process for dyeing of cellulosic-containing textiles with indigo  
IN Schnitzer, Georg; Suetsch, Franz; Schmitt, Michael; Kromm, Erich; Schlueter, Harald; Krueger, Rudolf

PA BASF A.-G., Germany

SO Ger. Offen., 4 pp.

CODEN: GWXXBX

PI DE 4310122 A1 941006

AI DE 93-4310122 930330

DT Patent

LA German

IC ICM D06P001-22

ICS D06P003-60

ICA C09B007-02; C09B067-28

CC 40-6 (Textiles and Fibers)

Section cross-reference(s): 61.

AB Title process comprises dyeing with an aq. soln. of leuco indigo which was prep'd. by catalytic hydrogenation and after exhaustion on the textile, converting to the pigment form in the usual way using air oxidn. The process eliminates sulfates in the waste water, reduces org. O demand, and provides optimum pH control.

ST dyeing leuco \*\*\*indigo\*\*\* catalytic \*\*\*hydrogenation\*\*\* ;  
cellulosic dyeing \*\*\*indigo\*\*\* catalytic \*\*\*hydrogenation\*\*\*

IT \*\*\*Hydrogenation\*\*\*  
(catalytic; of \*\*\*indigo\*\*\* in dyeing of cellulosic textiles)

IT Dyeing  
(of cellulosic-contg. textiles with leuco indigo prepd. by  
catalytic hydrogenation.)

IT 6537-68-4P, Leuco \*\*\*indigo\*\*\*  
RL: IMF (Industrial manufacture); NUU (Nonbiological use,  
unclassified); PREP (Preparation); USES (Uses)  
(catalytic \*\*\*hydrogenation\*\*\* product; process for dyeing of  
cellulosic-contg. textiles with indigo)

=>

1. 5,443,599, Aug. 22, 1995, Process for reduction of textile dyestuffs; Thomas Bechtold, et al., 8/623, 528, 598, 604, 650, 662 [IMAGE AVAILABLE]  
=> d ab

US PAT NO: 5,443,599 [IMAGE AVAILABLE]

L1: 1 of 1

ABSTRACT:

A process comprising the reduction of textile dyestuffs in an aqueous alkaline medium by means of a reducing compound which is a complex of an organic complexing agent and an iron (II)-salt. The iron (II)-salt is present in an amount sufficient to ensure the desired reduction of the dyestuff after a single oxidation.

=> s schnitzer,georg/in

L2 0 SCHNITZER,GEORG/IN

=> s 204/134/cclst

L3 0 204/134/CCLST

=> a 8/650/cclst

'A' IS NOT A RECOGNIZED COMMAND

=> s 8/650/cclst

L4 508 8/650/CCLST

=> s 204/73r/cclst

L5 0 204/73R/CCLST

=> 204/clas

'204' IS NOT A RECOGNIZED COMMAND

=> s 204/clas

L6 34846 204/CLAS

=> s 204/134/cclst

L7 0 204/134/CCLST

=> d acc cls 5244549

5,244,549 [IMAGE AVAILABLE]

2 CLASSIFICATIONS

ANS: 1

1. 205/688 OR

2. 205/691 XR

=> s 205/688/cclst

L8 137 205/688/CCLST

=> s l8 and indigo?

2463 INDIGO?

L9 1 L8 AND INDIGO?

=> d

1. 5,244,549, Sep. 14, 1993, Process for the reduction of dyes; Thomas Bechtold, \*\*205/688\*\*, 691 [IMAGE AVAILABLE]

=> s mediator#

L10 3478 MEDIATOR#

=> s l10 and ingido

0 INGIDO

L11 0 L10 AND INDIGO

=> s l10 and indigo

1718 INDIGO

L12 6 L10 AND INDIGO

=> d 1-6

1. 5,443,599, Aug. 22, 1995, Process for reduction of textile dyestuffs; Thomas Bechtold, et al., 8/623, 528, 598, 604, 650, 662 [IMAGE AVAILABLE]

2. 5,254,461, Oct. 19, 1993, Method of and apparatus for determining microorganism populations electrochemically; Gilson H. Rohrback, et al., 205/777.5; 422/75, 76, 77, 81; 435/25, 30, 32, 34, 39, 287.1; 436/150 [IMAGE AVAILABLE]

3. 5,244,549, Sep. 14, 1993, Process for the reduction of dyes; Thomas Bechtold, 205/688, 691 [IMAGE AVAILABLE]

4. 4,346,172, Aug. 24, 1982, Electrochemical process and apparatus to control the chemical state of a material; Mitchell R. Swartz, 205/701; 204/403; 205/703; 422/83, 98; 435/168, 173.1; 436/176 [IMAGE AVAILABLE]

5. 4,243,751, Jan. 6, 1981, Electrochemical process and apparatus to control the chemical state of a material; Mitchell R. Swartz, 435/168; 205/687, 766; 356/40, 41; 422/83, 98, 186; 435/173.2, 173.3, 173.8, 207; 436/176 [IMAGE AVAILABLE]

6. 4,139,348, Feb. 13, 1979, Electrochemical process and apparatus to control the chemical state of a material; Mitchell R. Swartz, 436/35; 128/653.1; 204/403; 205/777.5; 356/40, 41; 422/83, 108, 119; 435/4, 287.9, 288.7 [IMAGE AVAILABLE]

=> d hit 6

FILE WPAT;US5443599/PN;PRT FU IND

ELAPSED TIME ON ORBIT: 0.01 HRS.

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WPI 9605/UPEQ

DERWENT OFFERS NEW STATISTICAL ANALYSIS SOFTWARE - SEE NEWSDOC N249

SS 1 RESULT (1)

-1- (WPAT)

ACCESSION NUMBER  
SECONDARY ACCESSION  
TITLE

92-217107/26

C92-098367

Textile dyestuff redn. in aq. alkaline medium with  
ferrous salt - in complex form giving rapid redn. in  
single stage without pptn. e.g. in vat dyeing, metal  
complexes are stable in baths and printing inks  
E31 F06

DERWENT CLASSES  
PATENT ASSIGNEE

(VEFO-) VER FOERDERUNG FORSCHUNG & ENT; (VEFO-) VER  
FORDERUNG FORSCHUNG & ENTWICKLUNG

INVENTORS  
NUMBER OF PATENTS  
NUMBER OF COUNTRIES  
PATENT FAMILY

BECHTOLD T, BURTSCHER E,

3

16

W09209740-AI 92.06.11 (9226) 18p G D06P-001/22

EP-513291-AI 92.07.19 (9247) 18p G D06P-001/22

US5443599-A 95.08.22 (9539) 4p E C07B-031/00

G, E

LANGUAGE  
DESIGNATED STATE  
CITATIONS

\*US AT BE CH DE DK ES FR GB GR IT LU MC NL SE LI

02Jnl. Ref W09015182

PRIORITY  
APPLICATION DETAILS

90.12.03 90AT-002436

91.12.02 91WO-AT0123 91.12.02 91EP-920878 92.07.31

92US-916150 94.12.19 94US-358689

FILING DETAILS  
INT'L. PATENT CLASS.  
ABSTRACT

EP-513291 Based on W09209740

C07B-031/00 D06P-001/22

(W09209740-A)

In the redn. of textile dyestuffs (I) in aq. alkaline  
medium using a complex cpd. (II) with an Fe salt, the  
Fe-II salt (III) is used in the amt. needed for the  
required redn. of (I) in a single oxidn..

(III) is pref. Fe-II sulphate. (II) is pref.  
triethanolamine (IIA) or citric acid and may be added  
to a printing ink for printing the textile.

USE/ADVANTAGE - Useful in dyeing cellulose  
fibres with vat, sulphur and indigo dyestuffs. Addn.  
of (II) ensures adequate stability, redn. potential  
and reducing ion concn. and the oxidised salt remains  
in soln., avoiding the pptn. occurring in (III)-NaOH  
vats. Redn. is fast, complete redn. taking only  
seconds, which largely suppresses undesirable oxidn.  
by air and eliminates the need for excess reducing  
agent. The metal complexes are stable in dyebaths and  
printing inks. In addn., the redn. potential can be  
optimised by the choice of (II). (Dwg. 0/0)

DT Patent  
LA Russian  
IC C09B057-00  
CC 40-5 (Dyes, Fluorescent Whitening Agents, and Photosensitizers)  
AB Title compds. are prepd. by hydrogenating polycyclic quinones, thioindigos, or their halo derivs. in anhydr. pyridine over Raney nickel or a Pt-group metal or carriers and treating the product with a complex of SO<sub>3</sub> or ClSO<sub>3</sub>H with pyridine bases.  
ST sulfate leuco vat dye; \*\*\*hydrogenation\*\*\* catalyst \*\*\*vat\*\*\*  
\*\*\*dye\*\*\* ; nickel hydrogenation catalyst dye  
IT \*\*\*Hydrogenation\*\*\* catalysts  
(for \*\*\*vat\*\*\* \*\*\*dyes\*\*\* to leuco derivs.)  
IT Dyes  
(vat, leuco sulfate ester salts, manuf. of)  
IT 7440-02-0, uses and miscellaneous  
RL: USES (Uses)  
(Raney, catalysts, for \*\*\*hydrogenation\*\*\* of \*\*\*vat\*\*\*  
\*\*\*dyes\*\*\* )  
IT 7664-93-9DP, esters with leuco vat dyes, salts  
RL: IMF (Industrial manufacture); PREP (Preparation)  
(manuf. of)



a ali

ANSWER 1 OF 1 CA COPYRIGHT 1996 ACS

86:74408 CA

Salts of sulfate esters of leuco compounds of vat dyes

Rogovik, V. I.; Myslin, T. L.; Norchenko, V. N.; Arnol'dov, E. M.;

Rogovik, V. M.; Alekseev, V. I.

USSR

U.S.S.R.

From: Otkrytiya, Izobret., Prom. Obraztsy, Tovarnye Znaki 1976,

53(45), 226.

CODEN: URXXAF

SU 334850 761205

SU 70-1442099 700612

Patent

Russian

C09B057-00

40-5 (Dyes, Fluorescent Whitening Agents, and Photosensitizers)

Title compds. are prepd. by hydrogenating polycyclic quinones, thioindigos, or their halo derivs. in anhydr. pyridine over Raney

nickel or a Pt-group metal or carriers and treating the product with a complex of SO<sub>3</sub> or ClSO<sub>3</sub>H with pyridine bases.

sulfate leuco vat dye; \*\*\*hydrogenation\*\*\* catalyst \*\*\*vat\*\*\*

\*\*\*dye\*\*\* ; nickel hydrogenation catalyst dye

\*\*\*Hydrogenation\*\*\* catalysts

(for \*\*\*vat\*\*\* \*\*\*dyes\*\*\* to leuco derivs.)

Dyes

(vat, leuco sulfate ester salts, manuf. of)

7440-02-0, uses and miscellaneous

RL: USES (Uses)

(Raney, catalysts, for \*\*\*hydrogenation\*\*\* of \*\*\*vat\*\*\*

\*\*\*dyes\*\*\* )

7664-93-9DP, esters with leuco vat dyes, salts

RL: IMF (Industrial manufacture); PREP (Preparation)

(manuf. of)

=> s leuco (2a) indigo

3949 LEUCO

1723 INDIGO

L1 18 LEUCO (2A) INDIGO

=> d 1=18

THE UNIT OPTION IS NOT INTENDED FOR USE IN APS TEXT SEARCH

=> d 1-18

1. 5,378,246, Jan. 3, 1995, Indigo dye process; Sally Gurley, 8/625, 646, 653, 918 [IMAGE AVAILABLE]

2. 5,350,425, Sep. 27, 1994, Method of reducing vat dyes and the process of dyeing fabrics therein; David R. Carver, 8/465, 625, 651, 653; 585/266 [IMAGE AVAILABLE]

3. 4,740,453, Apr. 26, 1988, Silver halide photosensitive material containing a compound capable of releasing a photographically useful group; Koki Nakamura, et al., 430/505, 223, 264, 566, 955, 957, 959 [IMAGE AVAILABLE]

4. 4,699,627, Oct. 13, 1987, Indigo-dyeable polyester fibers and pretreatment of polyester to produce same; Bobby J. Bailey, 8/602, 581, 606, 653, 922 [IMAGE AVAILABLE]

5. 4,605,419, Aug. 12, 1986, 5,8-dihydroxy naphthalene-1,4-dione derivative and a hair dye composition containing the same; Masashi Kikuchi, et al., 8/426, 405, 406, 421, 650, 651, 653; 564/428 [IMAGE AVAILABLE]

6. 4,557,856, Dec. 10, 1985, Electrically conductive composition for electro-responsive recording materials; Nobuhiro Miyakawa, et al., 252/500, 518; 430/56, 96 [IMAGE AVAILABLE]

7. 4,392,141, Jul. 5, 1983, Image forming method; Eiichi Inoue, et al., 347/224; 346/135.1 [IMAGE AVAILABLE]

8. 4,286,017, Aug. 25, 1981, Heat-sensitive recording paper; Masato Nakamura, et al., 503/219; 427/150, 151, 152; 428/324, 327, 330, 331, 332, 338, 339, 913; 503/225 [IMAGE AVAILABLE]

9. 4,283,198, Aug. 11, 1981, Inert atmosphere indigo dyeing; John M. Fletcher, 8/653, 148, 918 [IMAGE AVAILABLE]

10. 4,214,031, Jul. 22, 1980, Conductive substrate for electrosensitive recording material; Nobuhiro Miyakawa, et al., 428/213; 427/58, 121, 342, 439, 443.1, 443.2; 428/246, 260, 262, 264, 265, 279, 322.7, 507, 514, 515

[IMAGE AVAILABLE]

11. 4,198,268, Apr. 15, 1980, Process for producing colored paper using granulated dye compositions; Alfred Frei, et al., 162/162; 8/524 [IMAGE AVAILABLE]

12. 4,118,183, Oct. 3, 1978, Process for the treatment of warp yarns; Eckhardt Godau, et al., 8/495, 107, 147, 151.2, 532, 597; 28/179 [IMAGE AVAILABLE]

13. 4,090,877, May 23, 1978, Photosensitive imageable composition containing a hexa-aromaticbimidazole, a leuco dye and an oxygen-sensitizing compound; Richard D. Streeper, 430/337, 342, 343 [IMAGE AVAILABLE]

14. 4,054,419, Oct. 18, 1977, Apparatus for conducting chemical reactions in the presence of two or more fluid phases; Glen C. Smith, et al., 422/211; 208/299 [IMAGE AVAILABLE]

15. 4,049,454, Sep. 20, 1977, Color radiography; Marcel Karel Van Doorselaer, et al., 430/7, 139, 363, 364, 369, 502, 505, 549, 567 [IMAGE AVAILABLE]

16. 4,035,244, Jul. 12, 1977, Electric recording process; Eiichi Inoue, et al., 347/163; 205/53, 54, 55; 430/41 [IMAGE AVAILABLE]

17. 3,974,041, Aug. 10, 1976, Image recording member with zeolitic water containing compounds; Masahiro Haruta, et al., 205/55, 54, 56; 346/135.1; 347/164 [IMAGE AVAILABLE]

18. 3,871,972, Mar. 18, 1975, Electrorecording sheet; Yoichi Sekine, 346/135.1; 205/53, 54, 55, 56; 427/150; 503/208, 211 [IMAGE AVAILABLE]  
=> d hit 8

US PAT NO: 4,286,017 [IMAGE AVAILABLE]

L1: 8 of 18

SUMMARY:

BSUM(23)

As the colorless or light-colored color-developing substance, there are used so-called leuco compounds capable of developing a color by reaction, such as triphenylmethane type leuco compounds, triphenylmethanephthalide type leuco compounds, fluorane type leuco compounds, laucoauramine type leuco compounds, spiropyran type leuco compounds, indoline type \*\*leuco\*\* compounds and \*\*indigo\*\* type \*\*leuco\*\* compounds. In the present invention, it is especially preferred to use triphenylmethane type and

fluorane type chromogenic substances.

=> 1d hit 2

'1D' IS NOT A RECOGNIZED COMMAND

=> d 2

2. 5,350,425, Sep. 27, 1994, Method of reducing vat dyes and the process of dyeing fabrics therein; David R. Carver, 8/465, 625, 651, 653; 585/266 [IMAGE AVAILABLE]

=> d hit 2

US PAT NO: 5,350,425 [IMAGE AVAILABLE]

L1: 2 of 18

#### ABSTRACT:

A process for reducing vat dyes, such as **indigo**, into their **leuco** form by placing a metal, such as aluminum, in water in the presence of a reduction facilitator to form a first solution after which a vat dye having the general structure  $O.dbd.C(--C.dbd.C--).sub.N C.dbd.O$  where  $N=1, 2$  or  $4$  is mixed with the first solution to form a dye solution where substantially all of the vat dye is reduced to its soluble leuco form having the general structure  $HO--C.dbd.(C--C).sub.N .dbd.C--OH$ . The process includes dyeing fabric in the dye solution. the reduction facilitator may be a base, for example that produced hydroxide ions in water, and the first solution as well as the dye solution can be a basic solution. The soluble leuco can be isolated from the dye solution and dissolved in a non-aqueous solution. Prior to mixing the vat dye in water, it may first be dissolved in a solvent miscible with water.

#### SUMMARY:

BSUM(2)

Generally, the field of this invention is the process of dyeing fabrics with a vat dye. Specifically, the present invention is related to the process of reducing vat dyes into their leuco form. More particularly, the present invention relates to the use of a metal reducing agent which is operative to reduce various vat dyes including **indigo** to their **leuco** form.

#### SUMMARY:

BSUM(14)

Still a further object of this invention is to provide an inexpensive reducing process for the reduction of dyes including **indigo** to their **leuco** form.

#### SUMMARY:

BSUM(20)

More specifically, the present invention is a process for reduction of **indigo** into its **leuco** form. This process has the following steps: placing aluminum in water in the presence of a reduction facilitator to form a first solution; and mixing indigo with the first solution to form a dye solution wherein substantially all of the indigo is reduced to the soluble leuco form.

SUMMARY:

BSUM(25)

Characteristically, vat dyes contain a chain of conjugated double bonds with two ketos groups in the end positions. Vat dyes have the following characteristic chemical structure: **STR6** The leuco form of a vat dye is **STR7** where the N is the same integer as the vat dye. The leuco form is a dihydro derivative of the vat dye as diols (vat acids). The leuco derivatives are very sparingly soluble in water. However, because the hydroxyl groups have an enol character, the vat acids are acidic ( $pK_{sub.a} = 9-11$ ). Thus, they dissociate in alkaline media and form soluble enolates. As is noted above, when  $n=1$ , the vat dye can be indigo. Indigo has the structure shown below: **STR8** The **leuco** form of **indigo** has the structure shown below: **STR9** The sodium hydroxide causes the relatively strong acid of the leuco form  $pK_{sub.a} = 9-11$  to ionize and become soluble in water. As can be seen in the drawing above, the carbon double bond has been broken to form the **leuco indigo**.

SUMMARY:

BSUM(27)

The process of the present invention reduces vat dyes to their leuco form. The preferred process reduces **indigo** to the **leuco indigo**. Generally, the process consists of placing metal in water in the presence of a reduction facilitator to form a first solution. The vat dye is then mixed into the first solution to form a dye solution. The dye solution contains the vat dye in its leuco form. Preferably, the process is employed with the vat dye indigo. The preferred method of reducing **indigo** to its **leuco** form is as shown. **STR10** Aluminum is placed in  $H_2O$  and a water/NaOH solution is added. This forms the first solution. The first solution is stirred and is preformed at room temperature  $25^{\circ}C$ . It should be noted other temperatures can be employed. Within two to five minutes, hydrogen gas will begin to evolve. Hydrogen gas must be vented to avoid any explosive reactions. After the hydrogen gas begins to evolve, the indigo is added to the mixture. The

indigo can be added lump wise or as small portions. Alternatively, the indigo can be dissolved in a solvent and added as a liquid. The selected solvent should not adversely effect the reducing reaction. After the indigo is added to the first solution, the dye solution is formed. The dye solution is stirred to permit the reducing reaction to go to completion. The completion of the reaction is evidenced by the lack of insoluble vat dye in the dye solution. Addition of aluminum may be necessary to complete a reaction that contains unreduced vat dye.

#### SUMMARY:

BSUM(37)

The fabric is immersed in the dye solution, which contains the reduced vat dye or **\*\*indigo\*\*** (in the **\*\*leuco\*\*** form), water and a base, preferably sodium hydroxide. "Fabric" is defined to include, without limitation, cotton, wools, and cotton blended with synthetics or other fibers such as rayon, polyacrylonitrile, and polyesters. The method of dyeing these fabrics is well known. They can include, without limitation, vat or continuous dyeing processes. There are processes that are called pad-steam, pad-batch, and long bath to name a few. Depending on which dyeing process is used, a variety of different temperature ranges can be employed. The reduced dye solution is usually between room temperature or 25.degree. C. to 110.degree. C. when the cloth is immersed therein. Preferably, the dye solution temperature is around 25.degree. C., though 60.degree. C. is common. Again, depending on which type of process for dyeing is employed, a variety of fabric process time can be employed. Immersion of the fabric in the dye solution frequently ranges from 30 minutes to 6 hours.

#### SUMMARY:

BSUM(40)

Depending on the type of fabric that is being dyed, I have found that the use of formic acid allows the oxidation of the leuco dye solution to proceed more rapidly in the air. After the fabric is dipped into the dye solution and allowed to drain, the fabric can be dipped into a dilute 0.1 M solution of formic acid instead of using acetic acid. The fabric is again allowed to drain. Then the fabric can be dipped into a rinse solution of water to remove residual and excess formic acid solution. Because indigo does not have a strong affinity for either wool or cotton fibers, the process for dyeing the fabric involves a repetition of the immersion process. Other dye solutions which use different vat dyes do not necessarily require a repetition of the dyeing process as they have more affinity for the fabric fibers than does the **\*\*leuco\*\*** form of **\*\*indigo\*\***.

DETDESC:

DETD(2)

An indigo dye stock solution was prepared. One gram of aluminum was added to one hundred ml of water. The solution was stirred, and five grams of fifty percent sodium hydroxide/H.sub.2 O was added. After a short period of time, approximately two to five minutes, the evolution of hydrogen was evident. Then one gram of solid indigo was added to the solution in a lump portion. The solid indigo was stirred for approximately one-half hour at 25.degree. C. The solution became de-colored from its original blue color of the indigo into a greenish-yellow color of the \*\*leuco\*\* form of \*\*indigo\*\* which is dihydroindigo. There was no overreduction of the indigo as the smell of fecal material was not present. There was 100% reduction as all the insoluble vat dye visibly went into solution.

CLAIMS:

CLMS(16)

16. A process for chemical reduction of \*\*indigo\*\* into its \*\*leuco\*\* form which comprises the steps of:

(a) placing aluminum metal in water in the presence of a reduction facilitator to form a first solution; and

(b) mixing indigo having this structure ##STR15## with the first solution to form a dye solution wherein substantially all of the indigo is reduced to the soluble leuco form having this structure: ##STR16##

=> s hydrogenate# (2a) indigo

42846 HYDROGENATE#

1723 INDIGO

L2 0 HYDROGENATE# (2A) INDIGO

=> s hydrogenate# (2a) vat dye#

42846 HYDROGENATE#

6119 VAT

85037 DYE#

955 VAT DYE#

(VAT(W)DYE#)

L3 0 HYDROGENATE# (2A) VAT DYE#

=> s hydrogenation (2a) (indigo or vat dye#)

35581 HYDROGENATION

1723 INDIGO

6119 VAT

85037 DYE#

955 VAT DYE#

(VAT(W)DYE#)

L4 0 HYDROGENATION (2A) (INDIGO OR VAT DYE#)

=> d hist

(FILE 'USPAT' ENTERED AT 07:17:20 ON 20 FEB 96)

L1 18 S LEUCO (2A) INDIGO

L2 0 S HYDROGENATE# (2A) INDIGO

L3 0 S HYDROGENATE# (2A) VAT DYE#

L4 0 S HYDROGENATION (2A) (INDIGO OR VAT DYE#)